Standard Operating Procedure for the Determination of n-hexane Extractable Material (oil and grease) by Solid Phase Extraction

1.0 Scope and Application

1.1 This method is applicable to the determination of n-hexane extractable material (HEM; oil and grease) in surface and saline waters and industrial and domestic aqueous wastes. Extractable materials that may be determined are relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases, and related materials. This method is a modification of the liquid-liquid extraction of oil and grease using n-hexane.

1.2 This method is not applicable to measurement of materials that volatilize at temperatures below approximately 85^0C. Petroleum fuels from gasoline through #2 fuel oil may be partially lost in the solvent removal operation.

1.3 Some crude oils and heavy fuel oils contain a significant percentage of materials that are not soluble in n-hexane. Accordingly, recoveries of these materials may be low.

1.4 This method is capable of measuring HEM in the range of 5 to 1000 mg/L and may be extended to higher levels by analysis of a smaller sample volume collected separately.

1.5 For HEM, the method detection limit is 1.4 mg/L and the minimum level of quantitation is 5.0 mg/L.

1.6 Modification of the method is permitted to overcome interferences or lower the cost of measurements, provided that all performance criteria in this method are met.

2.0 Summary of Method

2.1 One liter of sample is acidified to pH <2 and passed through an activated solid phase extraction disk. The disk is then extracted with n-hexane.

2.2 The hexane extract is dried by passing it through sodium sulfate into a pre-dried and weighed 40 mL vial. The hexane is evaporated and the vial is dried in an oven at 70^0 C and placed in a desiccator for 30 minutes after which it is weighed.

3.0 Definitions

The definitions below are specific to this method, but have been conformed to common usage as much as possible.

3.1 HEM: n-hexane extractable material that is extracted from a sample and determined by this method.

3.2 Laboratory blank: An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with samples. The laboratory blank is used to determine if analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

3.3 Matrix spike (MS) and matrix spike duplicate (MSD): Aliquots of an environmental sample to which known quantities of the analytes are added in the laboratory. The MS and MSD are prepared and /or analyzed like a field sample. Their purpose is to quantify any additional bias and imprecision caused by the sample matrix. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS and MSD must be corrected for background concentration.

3.4 May- This action, activity, or procedural step is neither required nor prohibited.

3.5 May not - This action, activity, or procedural step is prohibited.

3.6 Method Detection Limit: The lowest level at which an analyte can be detected with 99 percent confidence that the analyte concentration is greater than zero.

3.7 Minimum Level: The lowest level at which the entire analytical system gives a recognizable signal and acceptable calibration level for the analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all method specified sample weights, volumes, and cleanup procedures have been employed.

3.8 Must - This action, activity, or procedural step is required.

3.9 Ongoing precision and recovery standard: A laboratory blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and recovery.

3.10 Quality control sample: A sample containing analytes of interest at known concentrations. The QCS is obtained from a source external to the laboratory or is prepared from standards obtained from a different source than the calibration standards. The purpose is to check laboratory performance using test materials that have been prepared independently from the normal preparation process.

3.11 Shall - This action, activity, or procedural step is required.

3.12 Should - This action, activity, or procedural step is suggested, but not required.

3.13 Stock solution: A solution containing an analyte that is prepared using a reference material traceable to EPA, the National Institute of Standards and Technology (NIST), or a source that will attest to the purity and authenticity of the reference material.

4.0 Interferences

4.1 Solvents, reagents, glassware, and other sample-processing hardware may yield artifacts that affect results. Specific selection of reagents and purification of solvents may be required.

4.2 All materials used in the analysis shall be demonstrated to be free from interferences by running laboratory blanks.

4.3 Glassware is cleaned by washing in hot water containing detergent, rinsing with tap and distilled water, and rinsing with solvent or baking. Vials that will contain the extracted residue are dried in an oven at 105 - 115^0 C and stored in a desiccator.

4.4 Sodium sulfate has the potential to inflate results for HEM. Excessive water in the n-hexane extract can dissolve sodium sulfate during the drying process. Evidence of this is formation of a white solid on the side of the extract collection vial during evaporation of the n-hexane.

4.5 Interferences extracted from samples will vary considerably from source to source, depending upon the diversity of the site being sampled. For those instances in which samples are thought to consist of complex matrices containing substances (such as particulates or detergents) that may interfere with the extraction procedure, a smaller sample may need to be collected for analysis.

5.0 Safety

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level. It is suggested that personal hygiene monitoring of each analyst using this method be performed and that the results of this monitoring be made available to the analyst.

5.2 n-Hexane has been shown to have increased neurotoxic effects over other hexanes and some other solvents. OSHA has proposed a time-weighted average (TWA) of 50 parts-per-million (ppm); NIOSH concurs that an 8-hour TWA/permissible exposure limit (PEL) of 50 ppm is appropriate for n-hexane; and the American Conference of Governmental Industrial Hygienists (ACGIH) has published a threshold limit value (TLV) of 50 ppm for n-hexane. Inhalation of n-hexane should be minimized by performing all operations with n-hexane in an explosion-proof hood or well-ventilated area.

5.3 n-Hexane has a flash point of -23^0 C (-9^0 F), has explosive limits in air in the range of 1 - 7 percent, and poses a serious fire risk when heated or exposed to flame. n-Hexane can react vigorously with oxidizing materials.

5.4 Unknown samples may contain high concentrations of volatile toxic compounds. Sample containers should be opened in a hood and handled with gloves to prevent exposure.

5.5 This method does not address all safety issues associated with its use. A safe working environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method must be maintained by the laboratory. A reference file of material safety data sheets should be available to all personnel involved in these analyses.

6.0 Equipment and Supplies

Note: Brand names, suppliers, and part numbers are cited for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and materials other than those specified here, but demonstration of equivalent performance that meets the requirements of this method is the responsibility of the analyst.

6.1 Sampling equipment

6.1.1 1 liter glass sample collection bottles with PTFE-lined screw cap. Wash with detergent water, rinse with tap water, cap with aluminum foil and bake at 200-250^0 C for 1 hour minimum prior to use. Solvent rinse may be used in place of baking.

6.1.2 Bottles and liners must be lot-certified to be free of artifacts by running laboratory reagent blanks according to this method. If blanks from bottles and/or liners without cleaning or with fewer cleaning steps than required above show no detectable materials, the cleaning steps that have no effect may be omitted.

6.2 Equipment and supplies for sample extraction

6.2.1 Stainless-steel solid phase extraction manifold and accessories: 47 mm reservoir, base, screen, and clamps.

6.2.2 Oil and grease solid phase extraction disks.

6.2.3 Filter aid.

6.2.4 Low-range pH paper.

6.3 Equipment for drying extract

6.3.1 Solid phase extraction vacuum manifold chamber.

6.3.2 Sodium sulfate cartridges.

6.3.3 40 mL glass vials.

6.4 Equipment for solvent removal

6.4.1 Turbovap evaporator

6.5 Equipment for drying, cooling and weighing

6.5.1 Ovne - maintained at 70 $\pm 2^0$C.

6.5.2 Desiccator - Cabinet or jar type.

6.5.3 Analytical balance - Capable of weighing to the nearest 0.1 mg.

7.0 Reagents and Standards

7.1 Reagent water - water in which HEM is not detected at or above the minimum level of this method.

7.2 Hydrochloric acid or sulfuric acid - mix equal volumes of concentrated HCL and reagent water or 1 part concentrated H_2SO_4 and 3 parts reagent water to produce an approximate 6N solution.

7.3 n-Hexane - 85% minimum purity.

7.4 Acetone - ACS - residue <1 mg/L.

7.4 Sodium sulfate cartridges - 5.5 g.

7.5 Stearic acid - 98% minimum purity.

7.6 Hexadecane - 98% minimum purity.

7.7 Hexadecane/stearic acid (1:1) spiking solution - prepare in acetone at a concentration of 2 mg/mL each.

7.7.1 Place 200 $\pm$ 2 mg stearic acid and 200 $\pm$ 2 mg hexadecane in a 100 mL volumetric flask and fill to the mark with acetone. Solution may require warming for complete dissolution of stearic acid.

7.7.2 After the hexadecane and stearic acid have dissolved, transfer the solution to a 100 - 150 mL vial with fluoropolymer-lined cap. Mark the solution level on the vial and store in the dark at room temperature. The solution should be checked frequently for signs of degradation or evaporation and must be replaced after six months, or sooner if degradation has occurred.

7.7.3 Immediately before use, verify the level on the vial and bring to volume with acetone, if necessary. Warm to redissolve all visible precipitate. If there is doubt about the concentration, remove 10.0 $\pm$ 0.1 mL with a volumetric pipet, place in a tared weighing pan, and evaporate to dryness in a fume hood. The weight must be 40 $\pm$ 1 mg. If not, prepare a fresh solution as above.

7.8 Precision and recovery (PAR) standard - prepare by spiking 10.0 $\pm$ 0.1 mL of the hexadecane/stearic acid spiking solution into 950 - 1050 mL of reagent water to produce concentrations of approximately 20 mg/L each of hexadecane and stearic acid. The PAR standard is used for the determination of initial and ongoing precision and recovery. This solution should be checked frequently for signs of degradation or evaporation using the test noted in section 7.7.3, and must be replaced after six months, or sooner if degradation has occurred.

8.0 Sample Collection, Preservation, and Storage

8.1 Collect approximately one liter of representative sample in a glass bottle following conventional sampling practices, except that the bottle must not be pre-rinsed with sample before collection. To allow for potential QC failures, it is recommended that additional sample aliquots be collected. If analysis is to be delayed for more than four hours, adjust the sample pH to less than 2 with hydrochloric acid or sulfuric acid solution at the time of collection, and refrigerate at 0-4^0 C. To check pH, do not dip the pH paper in the container. Instead, use a stirring rod and dip it into the container and then touch it to the paper.

8.2 Collect an additional one or two aliquots of a sample for each set of twenty samples or less for the matrix spike and, if used, the matrix spike duplicate. For those circumstances requiring the collection of multiple aliquots of one sample, each aliquot is to be collected in either of the following ways: 1) collect simultaneously in parallel, if possible, or 2) collect as grab samples in rapid succession.

8.3 The high probability that extractable matter may adhere to sampling equipment and result in measurements that are biased low precludes the collection of composite samples for determination of oil and grease. Therefore, samples must be collected as grab samples. If a composite measurement is required, individual grab samples collected at prescribed time intervals must be analyzed separately and the concentrations averaged.

8.4 All samples must be analyzed within 28 days of the date and time of collection.

9.0 Quality Control

9.1 The minimum quality control requirements consist of an initial demonstration of

laboratory capability, ongoing analyses of standards and blanks as a test of continued performance, and analysis of a matrix spike (MS) to assess recovery. Laboratory performance is compared to established performance criteria to determine if the results of anlayses meet the performance characteristics of the method.

9.2 In recognition of advances that are occurring in analytical technology, certain options are permitted to improve separations or lower the costs of measurements, provided that all performance specifications are met. These options include alternate extraction and concentration devices and procedures such as solid-phase extraction, continuous liquid-liquid extraction and Kuderna-Danish concentration. Alternate determinative techniques, such as infrared spectroscopy or immunoassay, and changes that degrade method performance, are not allowed. If an analytical technique other than the techniques specified in EPA Method 1664A is used, that technique must have a specificity equal to or better than the specificity of the techniques in EPA Method 1664A for HEM in the sample of interest. Specificity is defined as producing results equivalent to the results produced by EPA Method 1664A for analytical standards and, where applicable, environmental samples, and that meet all of the QC criteria stated in EPA Method 1664A.

9.2.1 Each time a modification is made to EPA Method 1664A, the IPR test must be repeated to demonstrate that the modification produces results equivalent to or superior to results produced by EPA Method 1664A. If the detection limit of the method will be affected by the modification it must be demonstrated that the MDL (40 CFR Part 136, Appendix B) is less than or equal to the MDL in EPA Method 1664A or one-third the regulatory compliance limit, whichever is higher. If the modified method is to be used for compliance monitoring, the discharger/generator must also demonstrate that the modified method recovers an amount of HEM equivalent to the amount recovered by EPA Method 1664A on each specific discharge/waste stream. The tests required for this equivalency demonstration are given in section 9.2.3 of EPA Method 1664A.

9.2.2 Records of modifications made to EPA Method 1664A must be retained. (See section 9.1.2.2 of EPA Method 1664A for information on the records that must be retained.)

9.3 Initial demonstration of laboratory capability.

9.3.1 The MDL shall be determined per the procedure in 40 CFR 136, Appendix

B to establish the ability to detect HEM. An MDL less than or equal to 1.4 mg/L or less than 1/3 the regulatory compliance limit must be achieved prior to the practice of this method.

9.3.2 The following operations shall be performed to establish the ability to generate acceptable precision and accuracy.

9.3.2.1 Determine the concentration of HEM in four samples of the PAR standard according to the procedure beginning in section 11.

9.3.2.2 Using the results of the set of four analyses, compute the average percent recovery (X) and the standard deviation of the percent recovery (s) for HEM.

9.3.2.3 Compare s and X with the corresponding limits for initial precision and recovery in Table 1. If s and X meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If, however, s exceeds the precision limit or X falls outside the range for recovery, system performance is unacceptable. In this event, correct the problem and repeat the test.

9.3.3 To establish the ability of a modification of EPA Method 1664A to recover an amount of HEM equivalent to the amount recovered by EPA Method 1664A from a specific discharge/waste stream, proceed as follows:

9.3.3.1 Collect, extract, concentrate, and weigh the HEM in two sets of four aliquots of unspiked wastewater. One set of four wastewater aliquots is analyzed according to the protocol in Section 11 of EPA Method 1664A and the other set of four aliquots is analyzed using the modified method.

9.3.3.2 Calculate the average concentration of HEM for the set of results from EPA Method 1664A and for the set of results from the modified method. The average concentration using the modified method must be 78 to 114 percent of the average concentration produced by EPA Method 1664A for HEM. If not, the modified method may not be used. If the average concentration of the four results produced using EPA Method 1664A and the average concentration of the four results produced using the modified method are below the minimum level, and if the equivalency test of

the modified method is passed for spikes of reference standards into reagent water, the modified method is deemed to be equivalent to EPA Method 1664A for determining HEM on that specific discharge/waste stream.

9.3.4 A minimum of 5 percent of all samples from a given sampling site or, if for compliance monitoring, from a given discharge/waste stream must be spiked. The sample aliquot shall be spiked with the hexadecane/stearic acid spiking solution. A duplicate matrix spike (MSD) is recommended but not required.

9.3.4.1 If the concentration of HEM in the sample is being checked against a regulatory concentration limit, the spiking level shall be at that limit, at 1 to 5 times the background concentration of the sample or at the concentration of the OPR, whichever concentration is highest.

9.3.4.2 If the concentration of HEM in a sample is not being checked against a limit, the spike shall be at the concentration of the precision and recovery standard or at 1 to 5 times higher than the background concentration, whichever concentration is higher.

9.3.4.3 Analyze one sample aliquot out of each set of 20 samples from each site or discharge/waste stream to determine the background concentration of HEM.

9.3.4.4 If necessary, prepare a standard solution appropriate to produce a level in the sample at the regulatory compliance limit or at 1 to 5 times the background concentration. Samples containing high concentrations (>100 mg/L) of HEM will require a large volume of spiking solution for the MS (and MSD). If the concentration of HEM is expected to exceed 1000 mg/L, smaller sample volumes should be collected for the background measurement and MS (and MSD) so that the amount of HEM plus the amount spiked does not exceed 1000 mg/L.

9.3.4.5 Spike the additional sample aliquot(s) with the spiking solution and analyze the aliquot(s) to determine the concentration after spiking (A).

9.3.4.6 Calculate the percent recovery (P) of HEM in each aliquot using

the following equation:

$$P = \frac{100\ (A-B)}{T}$$

where:
A = Measured concentration of analyte after spiking
B = Measured background concentration of HEM
T = The concentration of the spike

9.3.4.7 Compare the percent recovery of the HEM with the corresponding QC acceptance criteria in Table 1. If the results of the spike fail the acceptance criteria, and the recovery of the QC standard in the ongoing precision and recovery test for the analytical batch is within the acceptance criteria in Table 1, an interference is present. In this case, the result may not be reported or used for regulatory compliance purposes and the laboratory must assess the potential cause for the interference. If the interference is attributable to sampling, the site or discharge/waste stream should be resampled. If the interference is attributable to a matrix problem, the method must be modified and the tests required in section 9.1.2 of EPA Method 1664A must be repeated as well as the analysis of the sample and the MS (and MSD, if performed). If the results of both the spike and the ongoing precision and recovery test fail the acceptance criteria, the analytical system is judged to be out of control, and the problem shall be identified and corrected, and the sample batch reanalzyed. All sample results must be associated with a valid MS (and MSD, if performed).

9.3.4.8 If a MSD is analyzed, compute the relative percent difference (RPD) between the MS and MSD (not between the two recoveries using the following equation:

$$RPD = \frac{[D_1 - D_2]}{(D_1 + D_2)/2} \times 100$$

where:
D_1 = Concentration of HEM in the sample
D_2 = Concentration of HEM in the duplicate sample

9.3.4.9 The relative percent difference for duplicates shall meet the

acceptance criteria in Table 1. If the criteria are not met, the analytical system is judged to be out of control, and the problem must be immediately identified and corrected, and the analytical batch reanalyzed.

9.3.5 Analyze laboratory reagent blanks to demonstrate freedom from contamination.

9.3.5.1 Extract and concentrate a reagent water blank initially and with each analytical batch using the same procedural steps as for samples.

9.3.5.2 If material is detected in the blank at a concentration greater than the minimum level, analysis of samples must be halted until the source of contamination is eliminated and a blank shows no evidence of contamination. All sample results must be associated with an uncontaminated method blank before the results may be reported for regulatory compliance purposes.

9.3.6 Analyze a precision and recovery standard to demonstrate that the analysis system is in control and that acceptable precision and accuracy is being maintained with each analytical batch.

9.3.6.1 Extract and concentrate a precision and recovery standard with each analytical batch according to the procedure.

9.3.6.2 Compare the recovery with the limits for ongoing precision and recovery in Table 1. If the recovery is in the range specified, the extraction, solvent evaporation, and weighing processes are in control and analysis of blanks and samples may proceed. If, however, the recovery is not in the specified range, the analytical process is not in control. In this event, correct the problem, re-extract the analytical batch, and repeat the ongoing precision and recovery test.

9.3.6.3 Results that pass the specification above should be added to IPR and previous OPR data and the QC charts should be updated to form a graphic representation of continued laboratory performance. A statement of laboratory data quality should also be developed for each analyte by calculating the average percent recovery (R) and the standard deviation of the percent recovery (s_r). Express the

accuracy as a recovery interval from R - $2s_r$ to R + $2s_r$.

9.3.8 It is suggested that a quality control sample (QCS) be obtained from a source different from the source for the hexadecane and stearic acid and used for verification of the concentration of HEM. The QCS should be analyzed monthly if routine analyses are performed and less frequently if anlayses are performed intermittently.

10.0 Calibration and Standardization

10.1 Calibrate the analytical balance at 2 mg and 1000 mg using class "S" weights.

10.2 Calibration shall be within $\pm$ 10% (i.e. $\pm$ 0.2 mg) at 2 mg and $\pm$0.5% (i.e. $\pm$5 mg) at 1000 mg. If values are not within these limits, recalibrate the balance.

11.0 Procedure

11.1 Bring the analytical batch of samples to room temperature.

11.2 Place about 1000 mL of reagent water in a clean beaker to serve as the laboratory blank.

11.3 Prepare the OPR (Section 7.8) using the PAR standard (Section 7.7).

11.4 Mark the sample bottle at the meniscus or weigh the bottle for later determination of sample volume.

11.5 Verify that the sample pH is < 2 using the following procedure: dip a glass stirring rod into the well mixed sample, withdraw the stirring rod and allow a drop of the sample to fall on or touch the pH paper.

11.5.1 If the sample is at neutral pH, add 5-6 mL of hydrochloric or sulfuric acid solution to the sample. If the sample pH is high, use a proportionally larger amount of HCL or H_2SO_4.

11.5.2 Replace the cap and shake thoroughly. Check the pH of the sample using the procedure described above. If necessary, add more acid and retest.

11.5.3 Add the appropriate amount of acid to the blank, OPR, MS (and MSD) to

adjust the pH of these solutions to <2.

11.6 Weigh the 40 mL vials to be used in extract collection. These vials should have been previously dried in an oven at 105-115 C for a minimum of 2 hours and stored in a desiccator.

11.7 Center the extraction disk on the base of the filtration apparatus and clamp the reservoir in place on top of the disk. If using glassware clamps, gently squeeze the jaws of the clamps to assure proper seating.

11.8 Add a level scoop of Filter Aid to the top of the disk so the bed depth is a uniform 1 cm.

11.9 Wash the disk and filter aid with 10-15 mL of n-hexane. Apply vacuum and pull about 1 mL through the disk and then let set for 2 minutes. After 2 minutes, then pull the rest through and let the disk dry. Repeat this step 2 more times.

11.10 Add 10-15 mL of methanol and pull 1 ml through the disk and wait for about 1 minute. Then pull enough through so there is just enough methanol to cover the filter aid.

11.11 Pour or decant the sample into the reservoir and apply vacuum. Decant as much liquid as possible before adding any of the sediment that may be present in the sample. Do not let the disk go dry. After the sample has all been added, allow the disk to dry by keeping the vacuum on for another 20 minutes.

11.12 Remove the whole glass filtration unit from the filtration apparatus and place a solvent rinsed 40 mL vial in position to collect the n-hexane extract from the disk. Replace the glass filtration unit and add 15 mL of n-hexane. Pull a small amount through and then let the disk soak for about 2 minutes. Pull the rest of the hexane through and keep adding hexane until the vial is almost full. Repeat for the rest of the samples.

11.13 Place the pre-weighed 40 mL vials in a solid phase extraction vacuum manifold under the delivery tips. Connect two sodium sulfate cartridges to the reservoir in series and place the assembly on the orifice of the vacuum manifold above the corresponding collection vial. Pour the hexane extract into the reservoir, apply vacuum and allow the extract to collect in the pre-weighed 40 mL vials.

11.14 Turn on the turbovap. Set the temperature of the water bath to 55^0C. It will take about 45 minutes to warm up.

11.15 After the turbovap warms up, place the 40 mL vials inside. Turn on the nitrogen and close the lid. Set the timer for 25 minutes.

11.16 After 25 minutes make sure all the hexane has been evaporated, if not, allow evaporation to continue for another 5-10 minutes. Inspect the vials for crystals. Crystal formation is an indication that sodium sulfate may have dissolved and passed into the vial. This may happen if the drying capacity of the sodium sulfate is exceeded or if the sample is not adjusted to low pH. If crystals are observed, redissolve the extract in n-hexane, quantitatively transfer through a filter into another tared 40 mL vial, and repeat the solvent evaporation procedure.

11.17 If there is no evidence of crystal formation in the vials, dry the vials for 30-45 minutes in an oven maintained at 70 ± 2^0C. Cool to room temperature in a desiccator and maintain in the desiccator for 30 minutes minimum. Remove with tongs and weigh immediately. Repeat the cycle of drying, cooling, desiccating, and weighing until the weight loss is less than 4% of the previous weight or less than 0.5 mg, whichever is less.

11.18 Fill the bottle that the sample came in to the mark with tap water, and then pour the water into a graduated cylinder that is capable of holding at least 1000 mL and then record this volume.

12.0 Data analysis, Calculations, and Reporting Results

12.1 Calculations

12.1.1 Calculate the concentration of HEM ("oil and grease") in the sample with the following equation:

$$\text{HEM (mg/L)} = \frac{\text{W (mg)}}{\text{V(L)}}$$

where:
W = Weight of extractable material. Subtract the vial tare weight from the total weight of the vial after drying.

V = volume of the sample in liters.

12.2 Reporting Results

12.2.1 The results should be reported in mg/L.

12.2.2 Results less than the minimum level (5 mg/L) should be reported as <5 mg/L.

13.0 Method Performance

13.1 For the liquid/liquid extraction using n–hexane True value=40 mg/L

$$\text{Mean} = \frac{38.4 + 37.3+37.7+37.6+38.5+41.1}{6} = 38.4$$

Standard Deviation(s)= 1.33

Variance (s^2)= 1.77

13.2 For the solid phase extraction true value = 40 mg/L

$$\text{Mean} = \frac{37.7+35.8+38.2+36.4+36+34.7+38.1}{7} = 36.7$$

Standard Deviation(s)= 1.39

Variance (s^2) = 1.92

13.3 Method Detection Limit

$$\text{Mean} = \frac{3.0+3.5+2.8+3.9+3.1+2.7+3.5}{7} = 3.2$$

Standard Deviation (s) = 0.43

Variance (s^2) = 0.18

14.0 Pollution Prevention

14.1 The solvents used in this method pose little threat to the environment when recycled and managed properly.

14.2 Standards should be prepared in volumes consistent with laboratory use to

minimize the volume of expired standards to be disposed of.

15.0 Waste Management

15.1 All Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions need to be complied with as they relate to any waste generated by this method. Compliance with all sewage discharge permits and regulations is also required.

15.2 Samples preserved with HCL or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

15.3 For further information on waste management consult "The Waste Management Manual for Laboratory Personnel," and "Less is Better: Laboratory Chemical Management for Waste Reduction," both available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036.

16.0 References

16.1 "Empore Extraction Disks for Oil and Grease Analysis", 3M Center, Building 220-9E-10, St. Paul, MN 55144-1000.

16.2 "Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, PB99-121949, February 1999, United States Environmental Protection Agency, Office of Water, Washington, DC 20460.

16.3 *Report of the Method 1664 Validation Studies*, April 1995. Available from the Sample control Center (operated by DynCorp I&ET), 6101 Stevenson Avenue, Alexandria, VA 22304, (703) 461-2100.

17.0 Tables

Table 1. Acceptance Criteria for Performance Tests

Acceptance Criterion	Limit (%)
Initial Precision and Recovery	

HEM Precision (s)	11
HEM Recovery (X)	83-101
Matrix spike/matrix spike duplicate	
HEM Recovery	78-114
HEM RPD	18
Ongoing precision and recovery	
HEM Recovery	78-114